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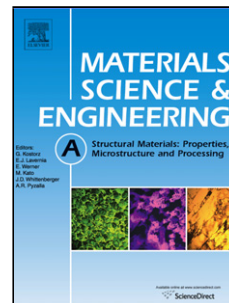
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- Comb samples used as reference samples in diffraction stress measurements on welds can retain significant macro-residual stress
- Measurements of the retained residual stress have been made in a comb sample extracted from a VPPA-welded aluminium plate
- A method is presented for the correction of the reference lattice parameter in the presence of retained residual stress

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# Problems in using a comb sample as a stress-free reference for the determination of welding residual stress by diffraction

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## Abstract

Precise measurement of a stress-free reference lattice parameter is vital in the determination of residual stress by diffraction techniques using  $\theta/2\theta$ -based analyses. For the evaluation of the residual strain profile in fusion-welded material, it is particularly important to correct the measured strain point-by-point by a stress-free reference for each spatial location across the weld. This is to take into account the compositional and microstructural variation across the weld caused by the thermal cycle of welding, as local changes in solute content of the parent alloy cause changes in the stress-free lattice parameter.

Although ideally such measurements should be obtained from small cubes machined from the weld, the use of a comb sample has previously been proposed as a macro-stress-free reference, with the assumption that each finger of the comb is of insufficient dimension to hold a macro-stress field. This paper presents an approach towards analysis of the stress-free reference values using a comb sample extracted from a Variable Polarity Plasma Arc (VPPA) welded plate. It is shown that there is inter-granular stress and retained macro-stress within the comb teeth, and an experimental approach to deal with the problem is proposed.

## 1 Introduction

Neutron and synchrotron X-ray diffraction are now widely used as techniques to determine residual strains – and hence stresses – deep inside engineering structures and components [1]. The average inter-planar spacing of a large number of grains

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inside a polycrystalline component is measured either for a single lattice reflection (by monochromatic radiation) or for multiple lattice reflections (by polychromatic radiation) and strain is determined by using the inter-planar spacing as an ‘atomic strain gauge’. This is based on the principle of Bragg diffraction, with the lattice spacing calculated using:

$$\lambda = 2d \sin \theta \quad (1)$$

where  $\lambda$  is the wavelength of the radiation used,  $\theta$  is half the diffraction angle  $2\theta$ , and  $d$  is the lattice spacing.

The strain is then calculated by:

$$\varepsilon = \frac{d - d_0}{d_0} \quad (2)$$

where  $d_0$  is the lattice spacing for the unstressed condition.

Accurate determination of strain, therefore, depends on measurement of the stress-free reference  $d_0$ . It has been observed that an error of 0.1% in  $d_0$  may lead to significant error (up to 1000  $\mu\varepsilon$ ) in strain calculation [2].

For an engineering component the stress-free reference can commonly be obtained in a number of ways [2], such as by using a powder sample or measuring at an assumed stress-free position within the material. However, the stress-free reference should reflect any change of lattice parameter owing to the mechanical and/or thermal processing history of the component: so for example, a powder reference should be subject to the same thermal history as the component being measured. More advanced methods are possible such as the use of Bragg edges [3, 4], or applying stress to a material to elucidate the stress-free parameter [5], but are rarely applied in practice.

In fusion welding, along the weld line the base metal melts and then re-solidifies. This is known as the fusion zone. The zone neighbouring the fusion zone experiences transient heating during each welding pass and this is known as the heat-affected zone (HAZ). The base metal in the HAZ does not melt, but the heating is enough to change the metallurgical condition of the base metal in the fusion and heat-affected zones.

The compositional variation that occurs during fusion welding due to the thermal cycle can change the stress-free reference lattice parameter. In most fusion welding processes a filler wire is introduced which also changes the composition in and around the fusion zone. The heat effect of welding also changes the state of

precipitates and cold worked structure. All these factors influence the stress-free lattice parameter. Therefore, to measure weld residual stress it is necessary to devise an appropriate method to determine the stress-free reference in order to correct the measured strains in the welded plate.

A common practice to measure the stress-free reference in a weld is to machine out a cube from an equivalent spatial location of the measured component, of insufficient dimension to hold an internal residual stress field [6-8]. Such cubes, if sufficiently small, can be assumed to be free of elastic stress. Inter-granular (type II) micro-stresses can remain, but should be the same as in the main component.

Machining of a comb sample across the weld to use as a stress-free reference is frequently used, with the assumption that each finger in a comb would be of insufficient dimension to hold an internal residual stress field [9, 10]. This is useful because machining and handling small cubes are difficult apart from the fact that set up of individual cube specimens in a neutron or synchrotron X-ray source can be prohibitively time consuming. Also a comparative study between the comb and cube methods revealed good agreement, although the comparative measurement was performed near the parent metal only [11].

In this paper, we report the evaluation of the stress-free reference lattice parameter observed during evaluation of the stress variation in a coupon plate sample welded by the Variable Polarity Plasma Arc (VPPA) process. A comb specimen was machined out by electro-discharge machining from the end of the plate, which allowed position-based correction of the measured strain in the welded plate. The comb specimen showed a distinct variation in the  $d_0$  values when measured by monochromatic neutron and synchrotron X-ray radiation for a single lattice reflection. A  $d$  vs.  $\sin^2 \psi$  experiment [12] was performed by synchrotron X-rays by measuring three additional tilts ( $\psi = 30^\circ, 45^\circ$  and  $60^\circ$ ) apart from  $\psi = 0^\circ$  and  $90^\circ$  which correspond to the longitudinal and normal directions respectively. This experiment was carried out to find the presence of any unrelieved elastic macro residual stress in the comb sample in the direction of its maximum dimension. The data showed the presence of unrelieved macro residual stress in the through thickness, normal, direction of the reference comb specimen. The reference lattice spacing data measured was then corrected accordingly

in light of the stress observed by the  $\sin^2\psi$  technique. The correction of the reference cell parameter based on the stress measured is based on the reasonable assumption that the unrelieved stress exists uniaxially in the direction of maximum dimension. Therefore, it provides an approximate correction of the reference lattice spacing measured in a comb sample.

The comb specimen was then measured by a white pulsed neutron beam at a spallation neutron source, where it is possible to determine the average cell parameter by refining all the peaks in the diffraction spectrum, which gives the average lattice parameter.

The study presented here is crucial in understanding that a comb sample may contain unrelieved residual stress field, and in order to have confidence in the measurements made using a comb sample it is necessary to characterize the macro-stress state that remains in the comb teeth.

## 2 Material

AA2024-T351 aluminium alloy, welded autogenously (i.e. without use of any filler metal), was studied here. Two AA2024 plates of length 500 mm, width 140 mm and thickness 12 mm, rolled and heat treated to the T351 condition, were welded together by a single-pass variable polarity plasma arc (VPPA) process [13]. Two sets of welded plates of 500 mm length were supplied. From each of the 500 mm long welded plates two pieces of 240 mm and 120 mm length along the welding direction were extracted, after sufficient discard from the ends to ensure that the samples were taken from a region of steady state welding conditions. The welding direction is the same as the rolling direction and considered as the longitudinal strain direction (LD), while the other two orthogonal directions along the width and the through thickness directions were termed the transverse (TD) and the normal strain directions (ND) respectively. A comb specimen with teeth dimensions of 2.7 mm  $\times$  2.7 mm  $\times$  12 mm was machined out from between the 240 mm and 120 mm long plates by electro-discharge machining using wire of 0.25 mm diameter. The reference comb sample was machined out  $\pm 60$  mm from the weld centre from a position in between the two plates of 240 mm and 120 mm length. A schematic diagram of the coupon welded plate and the stress-free reference comb sample is shown in figure 1, where an imaginary ABCD plane shows the measurement position in the coupon sample

described elsewhere [14]. The distance between the centres of two neighbouring fingers of the comb is approximately 3 mm. The objective of machining the comb sample is that the small dimension of each finger would be unable to hold any macro residual stress inside it. It can be seen from the figure that the dimension of each comb ‘finger’ in the normal direction is 12 mm, and 2.7 mm in the other two principal directions.

A hardness profile across the weld revealed a near-heat-affected zone (HAZ) up to  $\pm 12$  mm from the weld centre while the far HAZ is  $\pm 25$  mm from the weld centre [14].

### 3 Experimental Techniques

The residual stress in the coupon plate was determined by a combination of neutron and synchrotron X-ray diffraction. The plate was also studied after a skimming treatment to reduce the plate thickness, which will alter the stress field but does not require a different reference sample. The reference comb sample was measured under identical conditions as the welded plates, and the experimental details at the neutron and synchrotron X-ray sources used are given below. The comb sample was measured at all the sources detailed. When measurements were made in the comb teeth, a position towards the end of each tooth (typically  $\sim 4$  mm from the tooth end) was used.

#### 3.1 *Measurement by neutron diffraction: single wavelength source*

The as-welded coupon plate was measured on the D1A diffractometer at the reactor neutron source at the ILL, Grenoble, France. In this experiment strain measurements were made in the three principal directions, with corresponding measurements in the stress-free reference comb. An incoming beam size of  $2 \text{ mm} \times 2 \text{ mm}$  was used during the measurements, and a 1 mm collimator was used for the diffracted beam. The wavelength of the incident monochromatic beam was  $1.911 \text{ \AA}$ . The (311) lattice reflection was used, as this reflection is well-known to give good representation of the average lattice strain in the measured gauge volume [15]. The diffraction angle for this reflection at the stated wavelength was  $103^\circ$ . A schematic view of the comb set up is shown in figure 2a. Figure 2b shows the geometric gauge volume dimension along the different directions during measurement of the transverse strain direction. The gauge volume was carefully positioned inside each tooth of the comb by wall



scanning, as a partially-filled gauge would lead to the generation of pseudostrain because of spurious shifting of the diffraction peak position [16].

### 3.2 *Measurement with synchrotron X-rays*

The longitudinal and transverse strain directions of the as-welded and skimmed plate were measured at the ID31 beam line at the European Synchrotron Radiation Facility (ESRF), Grenoble, France. The smaller stress-free reference comb could be measured in all the principal directions, however measurement of the out-of-plane normal direction in reflection is not feasible in the plate sample as it would lead to a high path length for the beam, owing to the low scattering angle.

Synchrotron X-rays of 45 keV were used in the experiment, which corresponds to a wavelength of 0.2745 Å. The (311) plane of reflection was used for measurement which resulted in a diffraction angle  $2\theta$  of 12.5°. Measurement of the (311) plane allowed for comparison with the results obtained from the monochromatic neutron source.

A schematic set up of the side view during measurement of the normal direction is shown in figure 3. An incident beam of 2 mm × 2 mm was used. The vertical opening of the detector was kept at 0.5 mm. The resulting gauge volume shape and dimensions are also shown in figure 3. It can be seen that the instrumental gauge volume, which is defined by the intersection of the incident and diffracted beam, is elongated more than the dimension of the comb sample in the direction of the incident beam; therefore, the instrumental gauge volume extends beyond the irradiated volume of material. However, at beam line ID31 the detectors are preceded by Si (111) analyser crystals, and therefore the angle of diffraction is strictly defined by the diffracted beam from the sample, rather than the spatial position viewed by slits in front of the detectors. Hence partial immersion does not constitute a problem in the same way as it would have done when measuring at an instrument with no analyser crystal.

A  $d$  vs.  $\sin^2\psi$  experiment was performed to determine the presence of any stress component in the form of macro-stress in the normal direction, by measuring the comb sample in different  $\psi$  tilts. A schematic view of the  $\sin^2\psi$  experiment is shown in figure 4. The comb sample was measured in 5 different  $\psi$  tilts (0°, 30°, 45°, 60°, 90°) where 0° and 90° are measurements along the longitudinal and normal directions respectively. The stress was then evaluated using equation 3 by using the plane-

specific elastic constants, an approximate value of  $d_0$  and by determination of  $m$  from the linear regression of the  $d$  vs.  $\sin^2\psi$  data as shown in figure 5 [17].

$$\sigma_N - \sigma_L = \frac{E}{(1 + \nu) \times d_0} \times m \quad (3)$$

In this method of stress determination measurement of actual  $d_0$  is not necessary and an average  $d$ -spacing can be taken for  $d_0$  without introducing any significant error in the calculation [18]. A typical  $d$  vs.  $\sin^2\psi$  plot is shown in figure 5. The positive slope is indicative of a residual tensile stress. The stress measured in this method was then reasonably assumed to be the unrelieved stress in the normal direction as the longitudinal direction dimension ( $< 3$  mm) is insufficient to hold any elastic macro-stress field.

### 3.3 Measurement by pulsed neutrons

The stress-free reference comb was then measured by pulsed neutrons on the ENGIN diffractometer at the UK's ISIS spallation neutron source. At a spallation source, neutrons are produced in pulses and each pulse of neutrons has a range of wavelengths. Therefore, the diffraction spectrum comprises multiple peaks. For the ENGIN diffractometer at ISIS (now superseded by ENGIN-X), the detectors were positioned at a fixed angle and, depending upon the wavelength in a pulse satisfying Bragg's condition (equation 1), peaks for different lattice reflections are observed. The experiment by spallation neutrons was designed not only to see the  $d_0$  variation when multiple reflections are used, but also to observe the pattern for the other reflecting planes along the two principal directions.

A schematic top view for the comb measurement set up on ENGIN is shown in figure 6. The two detectors are separated mutually by  $180^\circ$ , at a fixed angle of  $\pm 90^\circ$  with respect to the incoming beam. The specimen is positioned so that two orthogonal strain directions are measured simultaneously by the two detectors. The longitudinal and transverse directions of the stress-free comb were measured simultaneously as shown in figure 6.

The average stress-free parameter was obtained by Pawley refinement [19] of the entire diffraction spectra using GSAS software [20]. Individual peaks are also refined using GSAS software. The comparison between the average stress free lattice

parameter and lattice spacing from individual peaks reveals the anisotropy between different crystallographic directions.

## 4 Results

The reference parameter variation, as measured in the comb specimen, for the three principal directions as observed by monochromatic neutrons at the ILL is shown in figure 7. Although near the weld the stress free lattice parameters show similar variations in the different directions, in the parent metal a distinct difference is observed between the different directions.

A similar  $d_0$  variation was observed when measured by monochromatic synchrotron X-rays at ESRF (figure 8): this would be expected as at both sources the (311) lattice reflection was measured. The measurements at the monochromatic sources clearly indicate a difference in variation between the longitudinal direction and the other two principal directions.

As explained before the stress variation measured using the  $d$  vs  $\sin^2 \psi$  technique was uniaxially present in the normal direction and is shown in figure 9. A macro residual stress of about 45 MPa is observed near the parent metal while near the centre of the weld within a region of  $\pm 20$  mm the stress magnitude is less than 10 MPa. These stress values are sufficiently high to cause error in the final weld stress calculations, and it was therefore necessary to account for their presence in the evaluation of the  $d_0$  values for the weld. The results also highlight a potential problem in the use of comb samples for  $d_0$  calculation, as the comb dimensions can allow for significant residual stress to be retained. The method of measuring in the comb tooth close to the free end [10], where the stress will be relaxed approaching the free surface, would be likely to provide more accurate results, but removes the advantage of the comb sample of rapid counting by opening up the gauge volume along the length of the comb teeth.

Additionally, this can lead to measurements being made away from the weld bead in certain geometries, so giving increased error in the  $d_0$  determination as the compositional variation of reference cell parameter would not be accounted for precisely because the strain measurement was performed at the central through-thickness line.

With this stress data the lattice parameter measured in the normal direction can be corrected [21] for the unrelieved stress as shown in equation 5.

$$a_{0,ND} = \frac{a_{ND}}{1 + \frac{\sigma_N}{E}} \quad (5)$$

where  $a_{0,ND}$  is the lattice parameter after correction and  $a_{ND}$  is the measured lattice parameter.  $\sigma_N$  is the stress measured in the normal direction by the  $\sin^2\psi$  technique and  $E$  is the Young's modulus.

The longitudinal and transverse lattice parameters are also corrected for Poisson strain as shown in equation 6.

$$\left. \begin{aligned} a_{0,LD} &= a_{LD} \left(1 + \frac{\nu}{E} \sigma_N\right) \\ a_{0,TD} &= a_{TD} \left(1 + \frac{\nu}{E} \sigma_N\right) \end{aligned} \right\} \quad (6)$$

where  $a_{0,LD}$  and  $a_{0,TD}$  are the lattice parameters after correction in the transverse and longitudinal directions respectively,  $a_{LD}$  and  $a_{TD}$  are the measured lattice parameters in the longitudinal and transverse directions, and  $E$  and  $\nu$  are the elastic constants, the Young's modulus and Poisson's ratio respectively.

The correction of the measured reference lattice parameter of each position was done based on the stress measured in the corresponding finger of the comb sample. The correction leads to a change in strain magnitude of about 600  $\mu$ strain in the normal direction and 215  $\mu$ strain in the longitudinal and transverse directions near the parent metal. Fig. 10 shows the reference lattice parameter variation after correction. Note that the longitudinal and normal direction lattice parameters show good agreement after the correction, although there is still a difference for the transverse direction. The reference cell parameter variation for the multiple diffraction spectra observed at ISIS is shown in figure 11. The reference cell parameter variation in the longitudinal and transverse directions show similar trends in this case. This strongly suggests that the difference in variation observed when measured by monochromatic radiation is due to inter-granular strains between the longitudinal and transverse strain directions. This can be investigated further by examination of the behaviour of the individual diffraction peaks in the spectra. The reference lattice parameter variation obtained from refinement of individual peaks of the diffraction spectra at ISIS is shown in figure 12. It can be seen that the individual lattice reflections show different trends in the two principal directions; even though the average parameters obtained by refinement of the entire diffraction spectra are similar. Therefore, it can be inferred

that the differences in trend between the longitudinal and transverse strain direction is due to inter-granular strains between the different directions. This is entirely plausible, as the plates are produced by a rolling route involving extensive plastic deformation, and aluminium is plastically anisotropic [22].

The anisotropy strain from previous plastic deformation present in a crystal lattice would lead to line broadening [23]. Due to its very high peak-width resolution ( $\sim 0.003^\circ$ ) synchrotron diffraction peaks are ideal for analysis of peak broadening. Figure 13 shows the centreline peak-width analysis of the reference comb. The higher peak-width near the parent plate in the transverse direction suggests the presence of plastic anisotropy strain.

## Conclusions

1. The variation in stress-free lattice parameter around a VPPA weld in 2024 aluminium has been determined using neutron and synchrotron X-ray diffraction. A comb sample was machined from a welded sample to provide the reference values, with the assumption that the small dimensions of the comb teeth would relax any residual stress present. However, retained macro residual stress was observed in the normal direction of the reference comb sample, which is the direction of its maximum dimension (12 mm).
2. The stress magnitude along the direction of maximum dimension in the comb teeth was determined by the  $\sin^2 \psi$  technique, and the measured reference comb cell parameters were approximately corrected for the unrelieved macro-stress.
3. The presence of plastic inter-granular strain was observed in the transverse direction. Analysis of single peaks from multi-peak spallation source diffraction spectra showed the effects of anisotropy between the different principal directions. Peak-width analysis of synchrotron X-ray peaks also showed line broadening in the transverse direction near the parent plate.
4. The results show that comb samples should not be used to provide a stress-free reference without corresponding measurement of retained macro-stress. The proposed method of measuring only near the end of a tooth reduces the advantage of using the full tooth length to minimize count times, and may lead to errors in the position of the measured reference relative to the weld bead.

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## Figure captions

Figure 1 – Schematic diagram of the welded plate measured, and the comb sample machined out from the end of the plate. LD = longitudinal direction; TD = transverse direction; ND = normal direction.

Figure 2. a) Schematic set up for measurement of the comb on the D1A diffractometer, ILL; b) shows the actual gauge volume shape and dimensions due to the diffraction geometry during measurement of transverse direction lattice parameter. The PSD is the position-sensitive neutron detector.

Figure 3. a) Side view of experimental set up during measurement of the normal strain direction at ID31, ESRF; b) The instrumental gauge volume dimension during measurement of the normal direction strain (Note: The actual irradiated volume is less than the instrumental gauge volume as the instrumental gauge volume was elongated in the direction of the incident beam)

Figure 4 – Experimental set up of the  $\sin^2\psi$  experiment. The angle  $\psi$  at different tilts is the angle subtended by the longitudinal direction vector with the incoming beam

Figure 5 – A typical  $d$  vs.  $\sin^2\psi$  curve. The slope of this curve was used to compute the unrelieved elastic macro stress in the normal direction of the comb specimen.

Figure 6 – Experimental set-up for measurement of the comb sample on the ENGIN diffractometer at ISIS. The transverse direction is measured at the left detector and the longitudinal direction is measured by the right detector.

Figure 7 – The stress free reference parameter variation along the three principal directions measured at ILL

Figure 8 – The stress free reference parameter variation along the three principal directions measured at ESRF. Note that a complete measurement was performed in one side of the weld only, while up to 20 mm was measured on the other side. The overall profile shown is produced by mirroring the data from one side based on the assumption that there would not be any significant variation. The error bar from

experimental uncertainty has been removed for clarity. The maximum uncertainty in peak positioning was about 15  $\mu\epsilon$

Figure 9 – Stress variation in the normal direction as observed by the  $\sin^2\psi$  technique

Figure 10 – The stress-free reference parameter variation along the three principal directions after correction by the macro-stress measured using the  $\sin^2\psi$  data

Figure 11 – Reference lattice parameter variation in the longitudinal and transverse directions as measured at ISIS. A total of 11 teeth spanning a distance of 78 mm on one side of the weld and 12 mm in the other side of the weld were measured. In figure 11 some of the data measured in one side of the weld have been mirrored into the other side assuming symmetry.

Figure 12 – Reference lattice parameter variation in the longitudinal and transverse directions obtained from individual peak refinement of the diffraction spectra at ISIS.

Figure 13 – Peak-width variation in the reference comb sample. Peak-widths are measured from diffraction data at ID31, ESRF.

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Figure 1

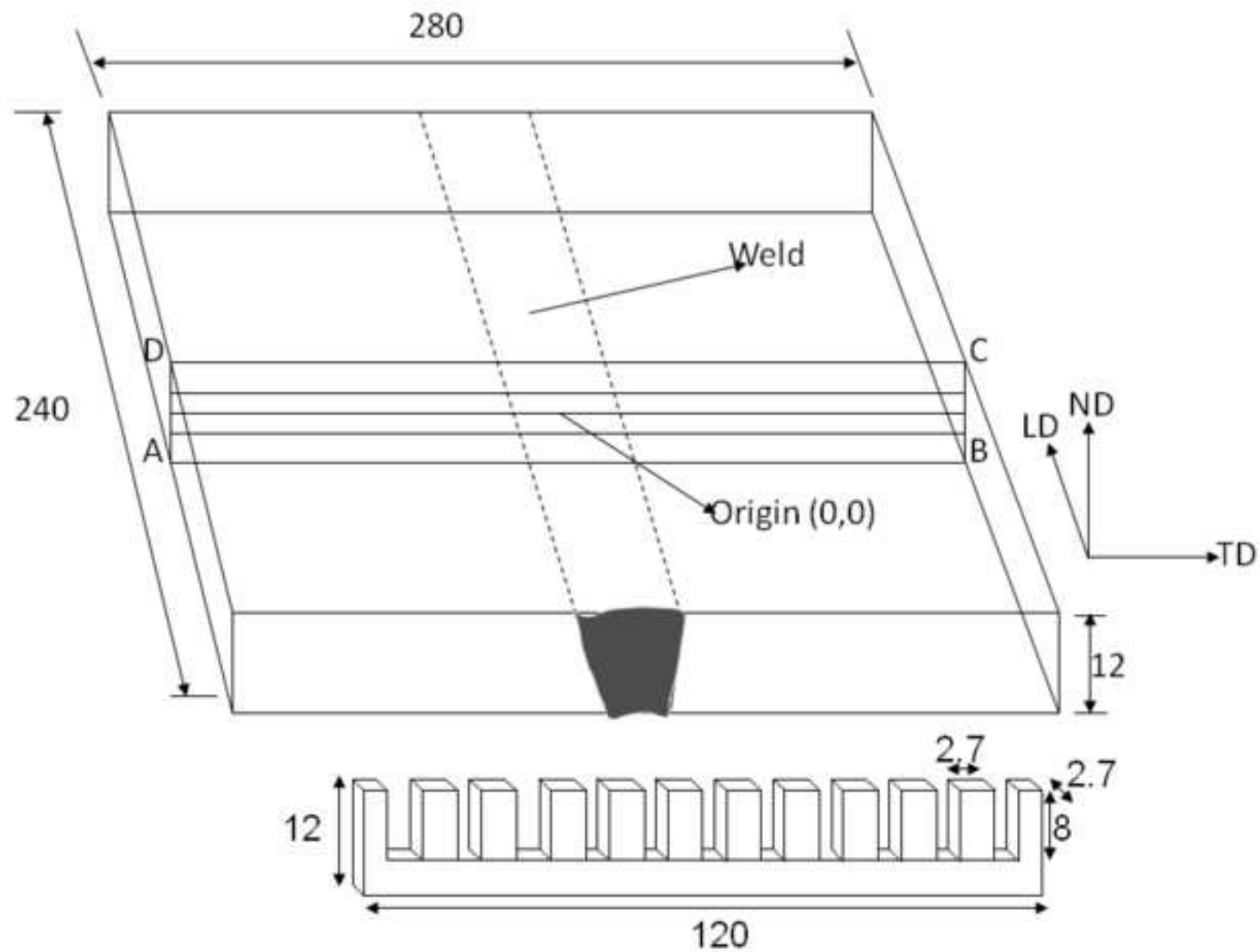


Figure 2

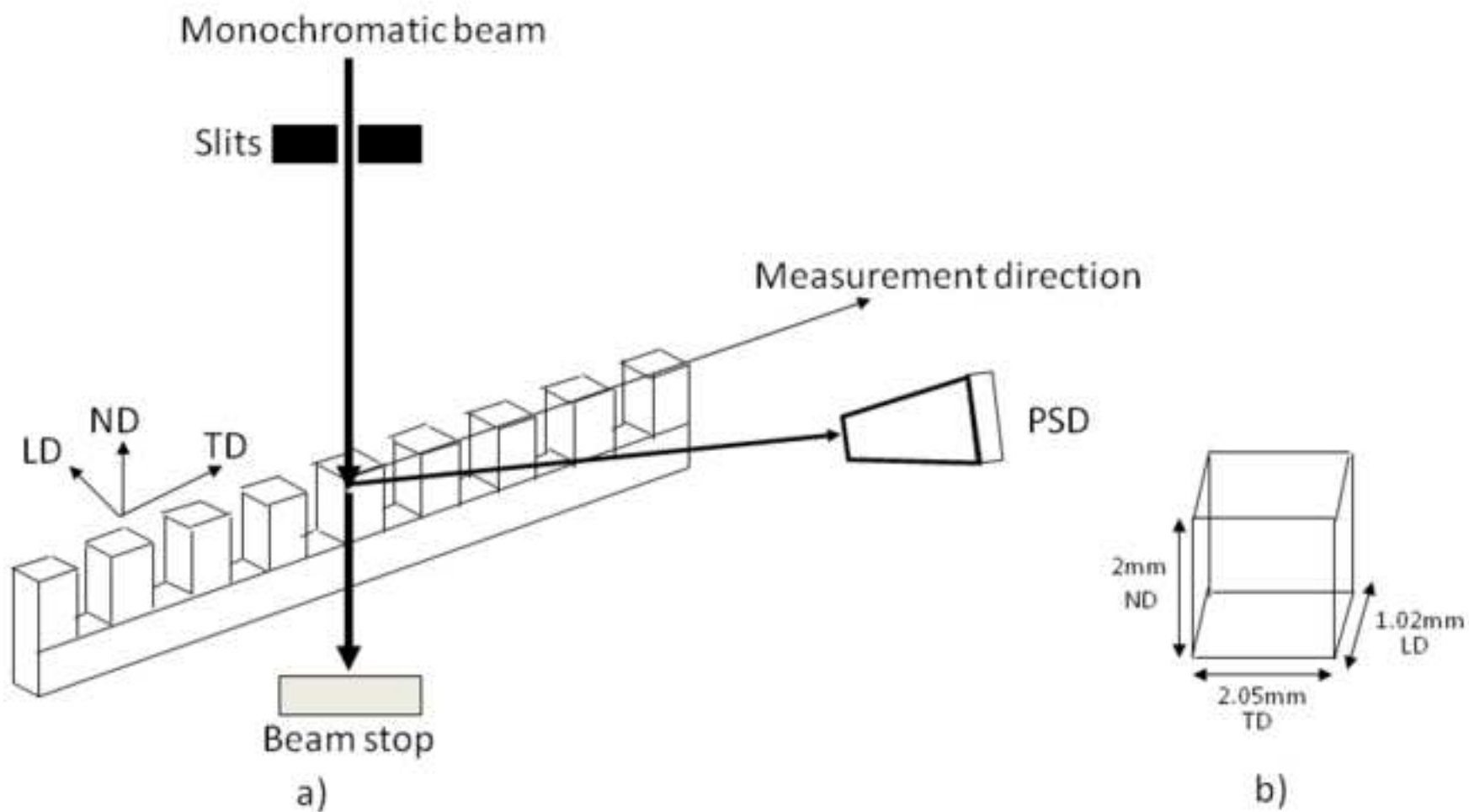


Figure 3

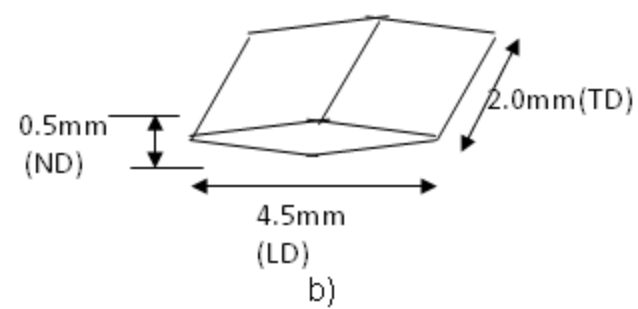
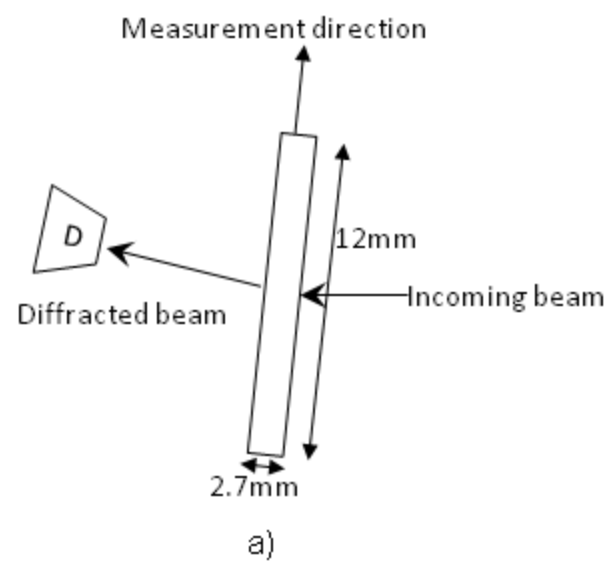


Figure 4

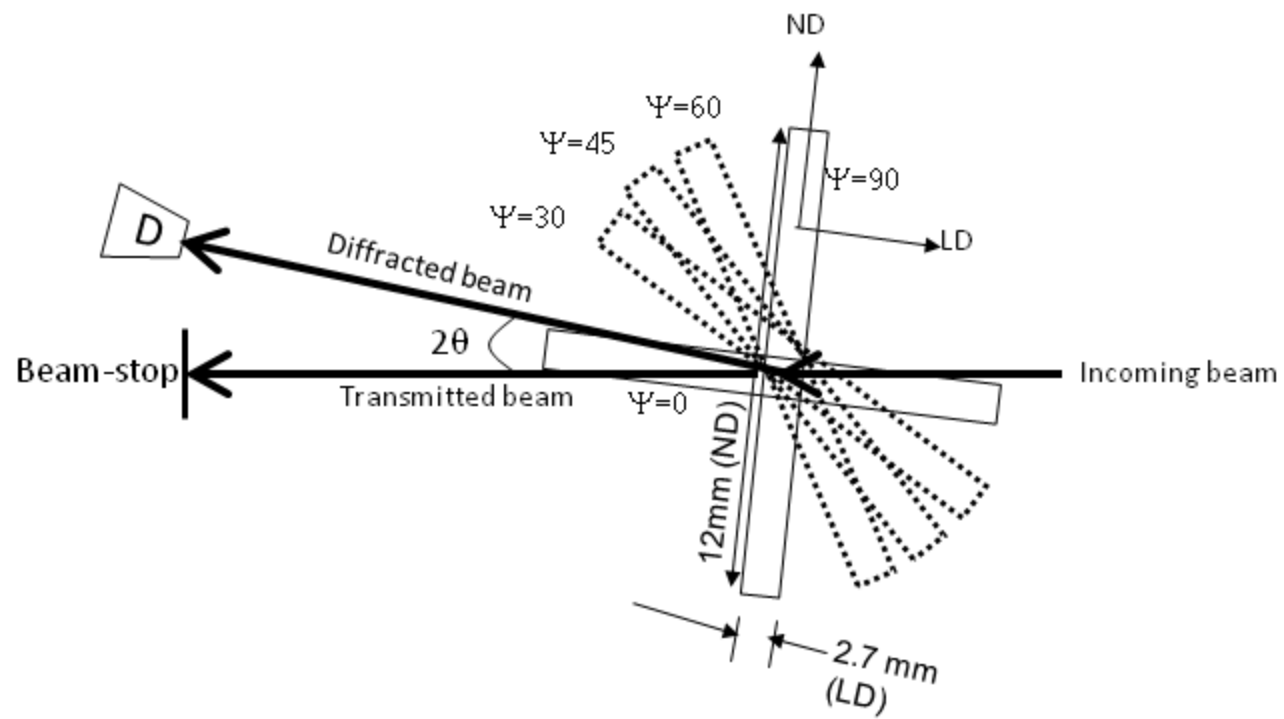


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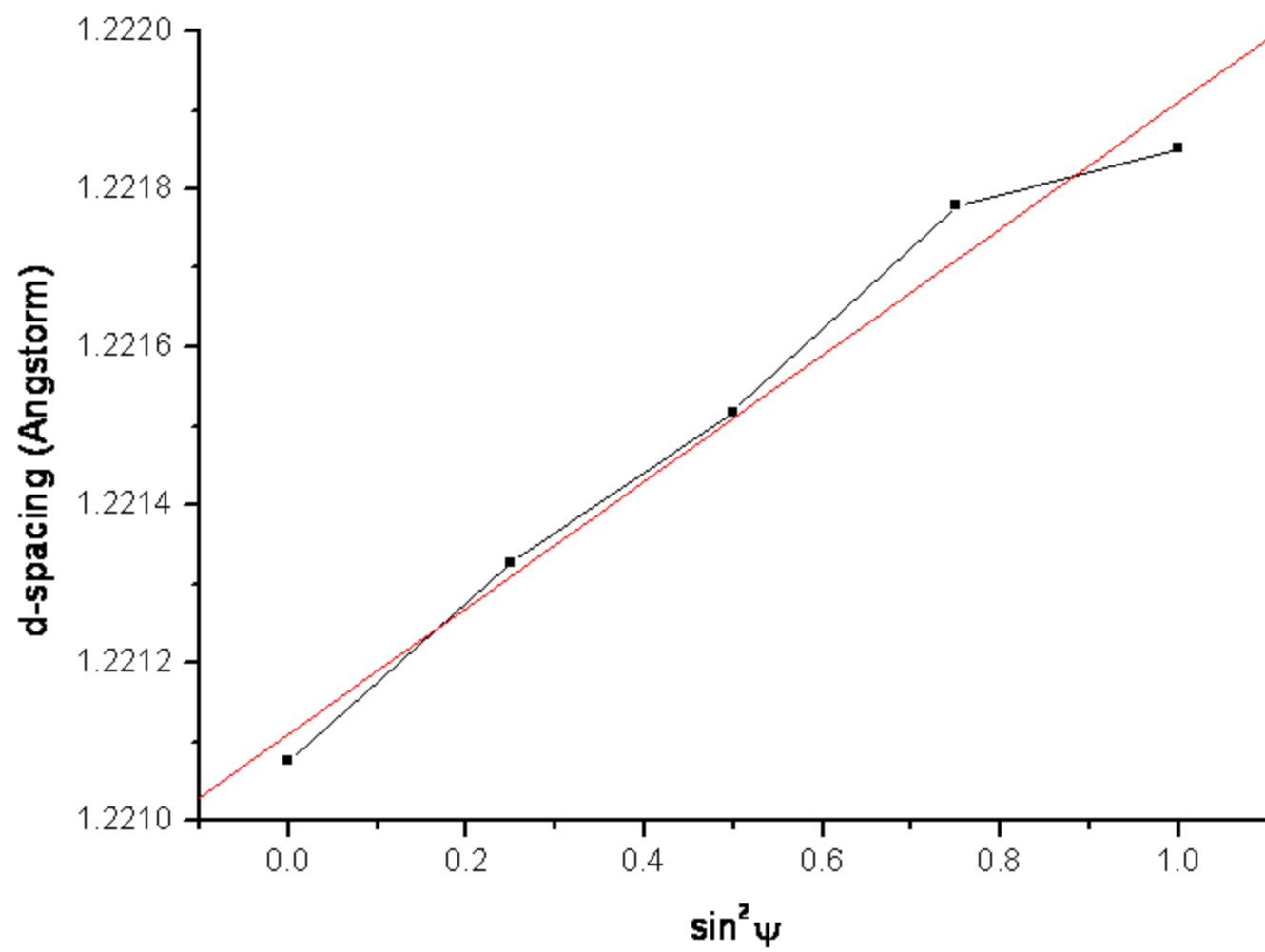


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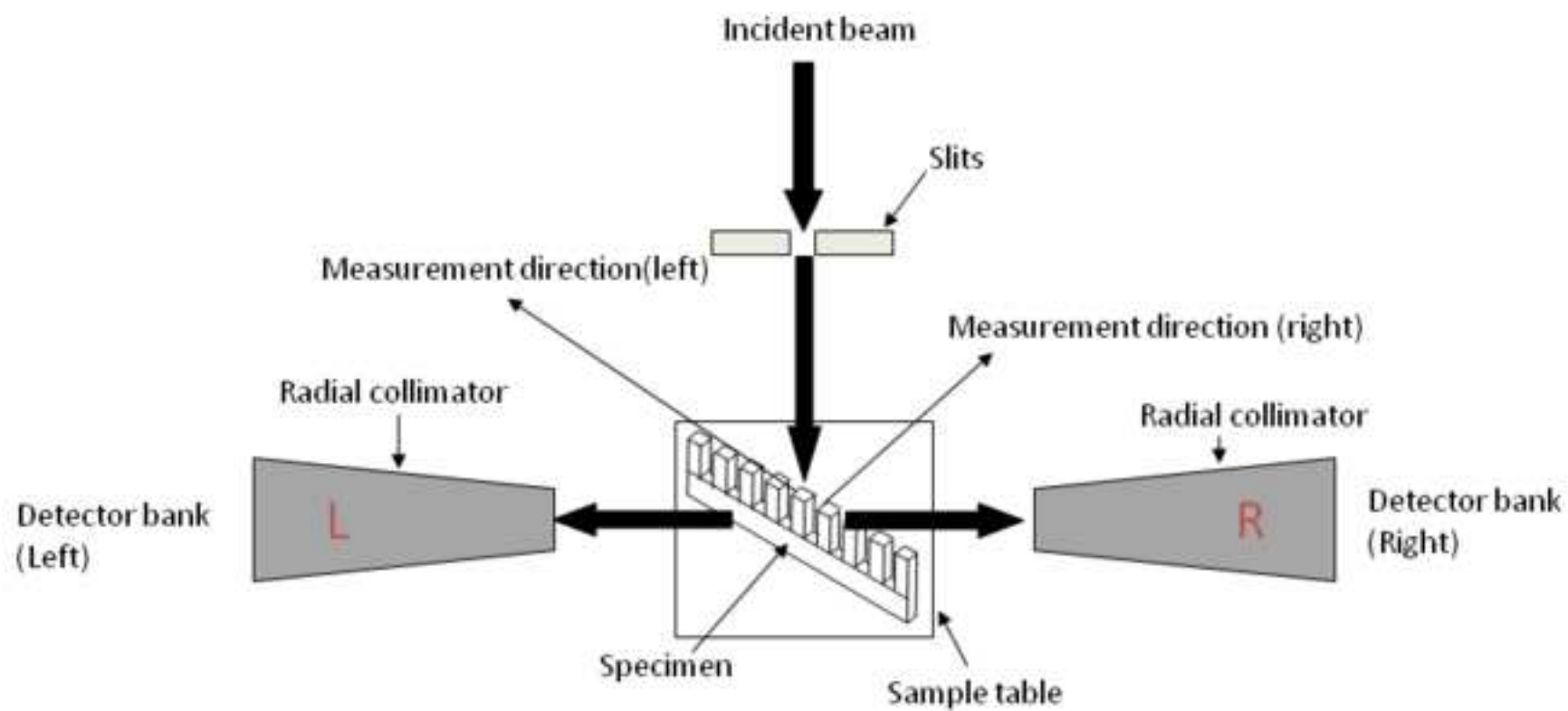


Figure 7

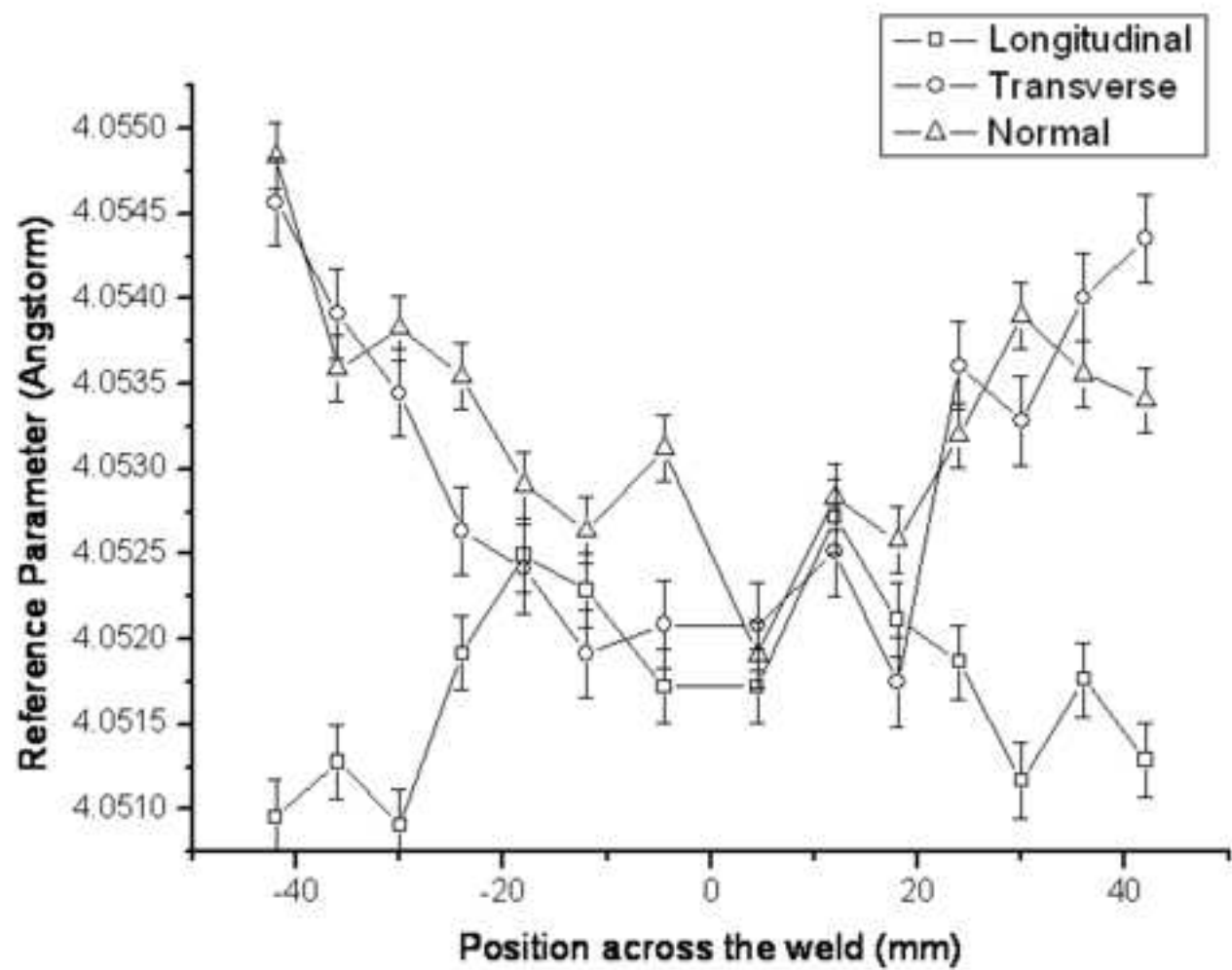




Figure 8

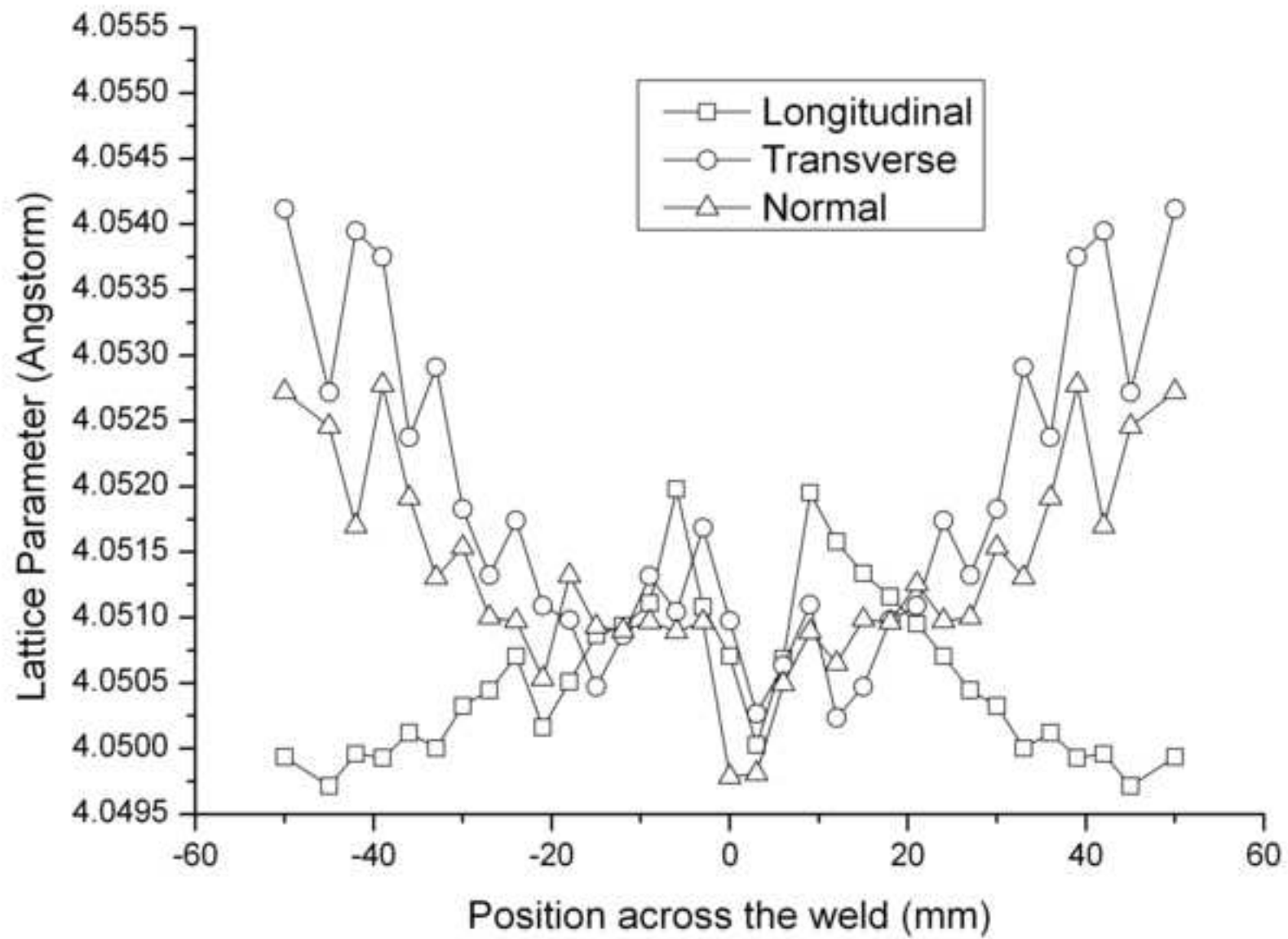


Figure 9

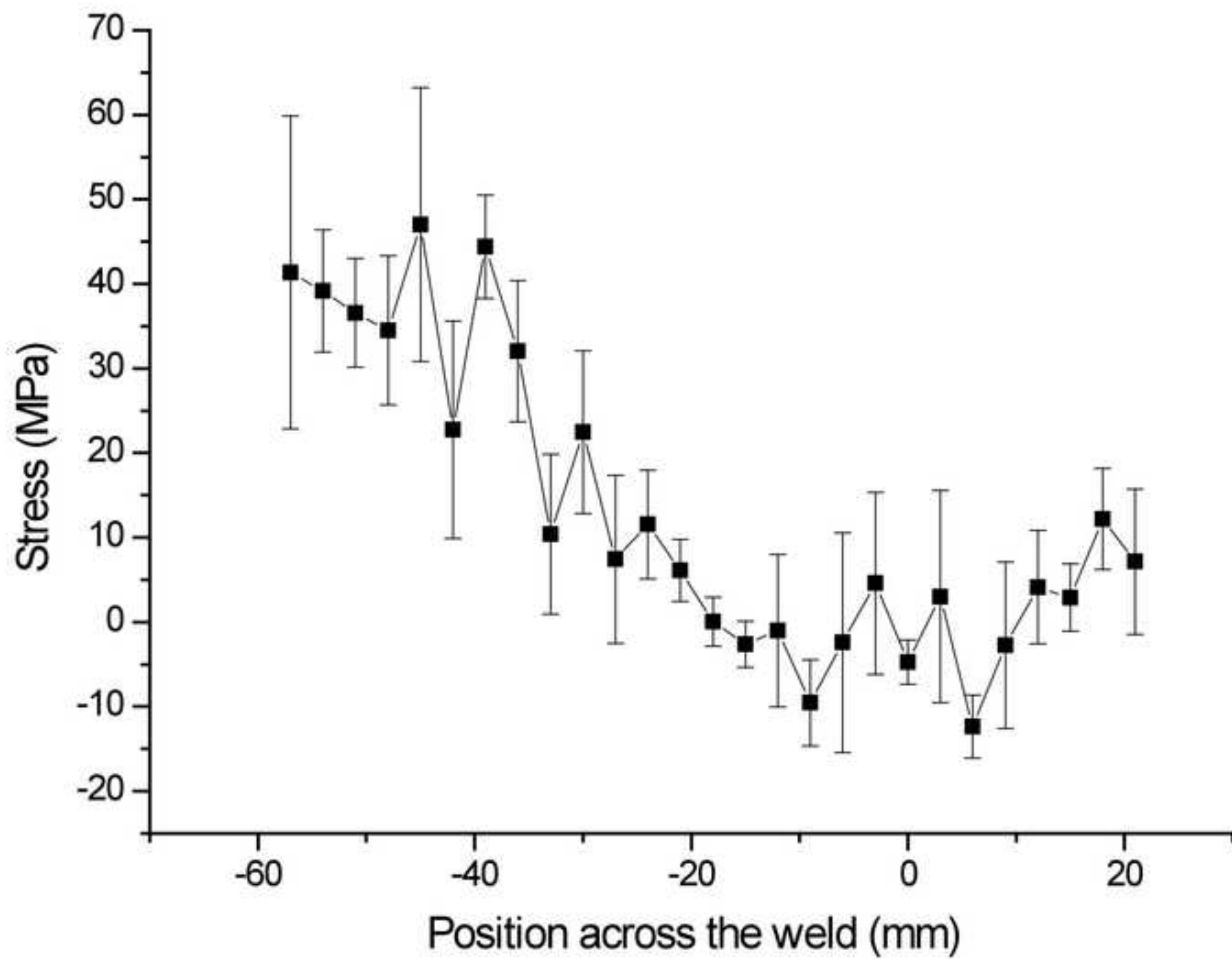


Figure 10

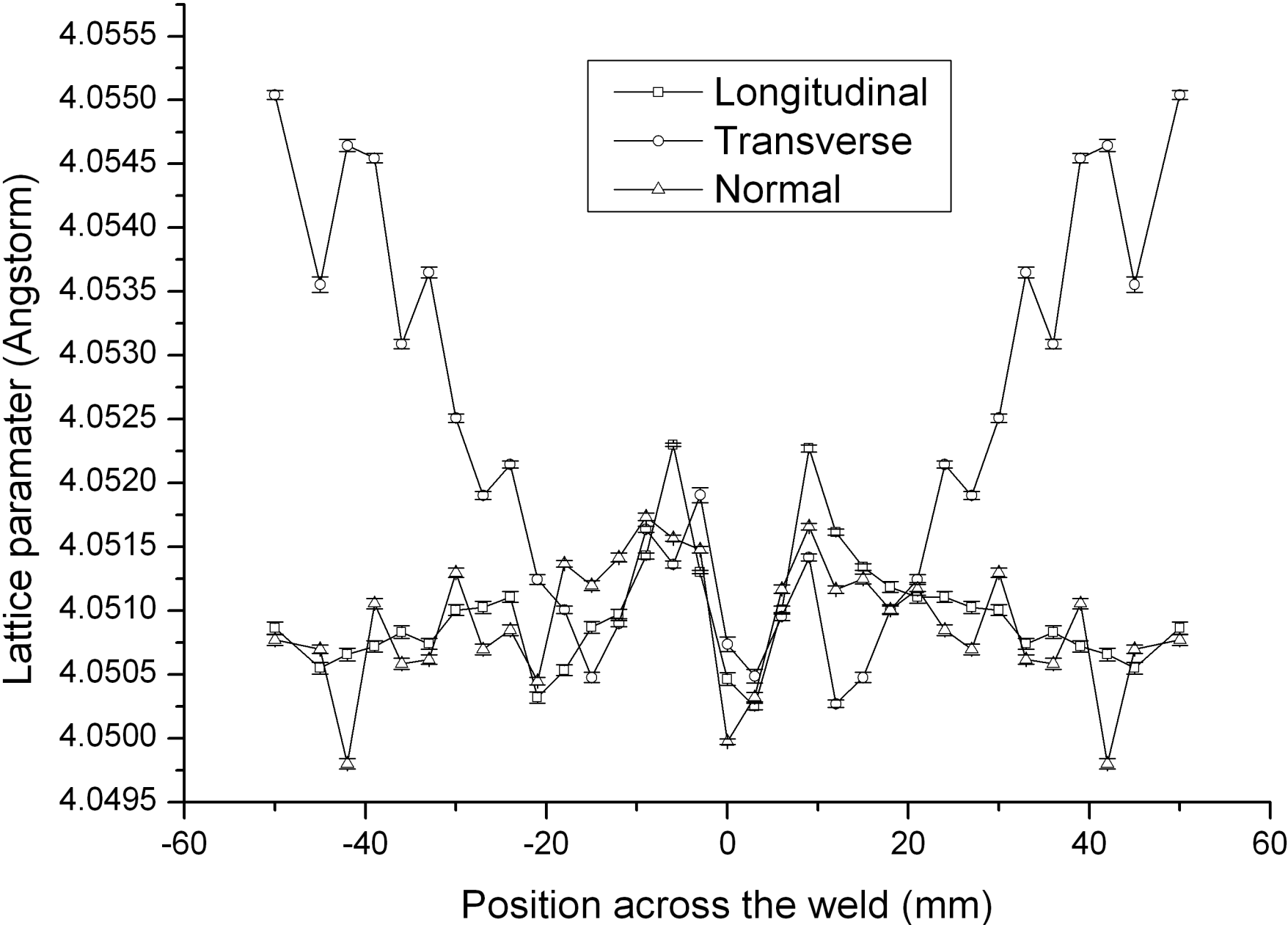


Figure 11

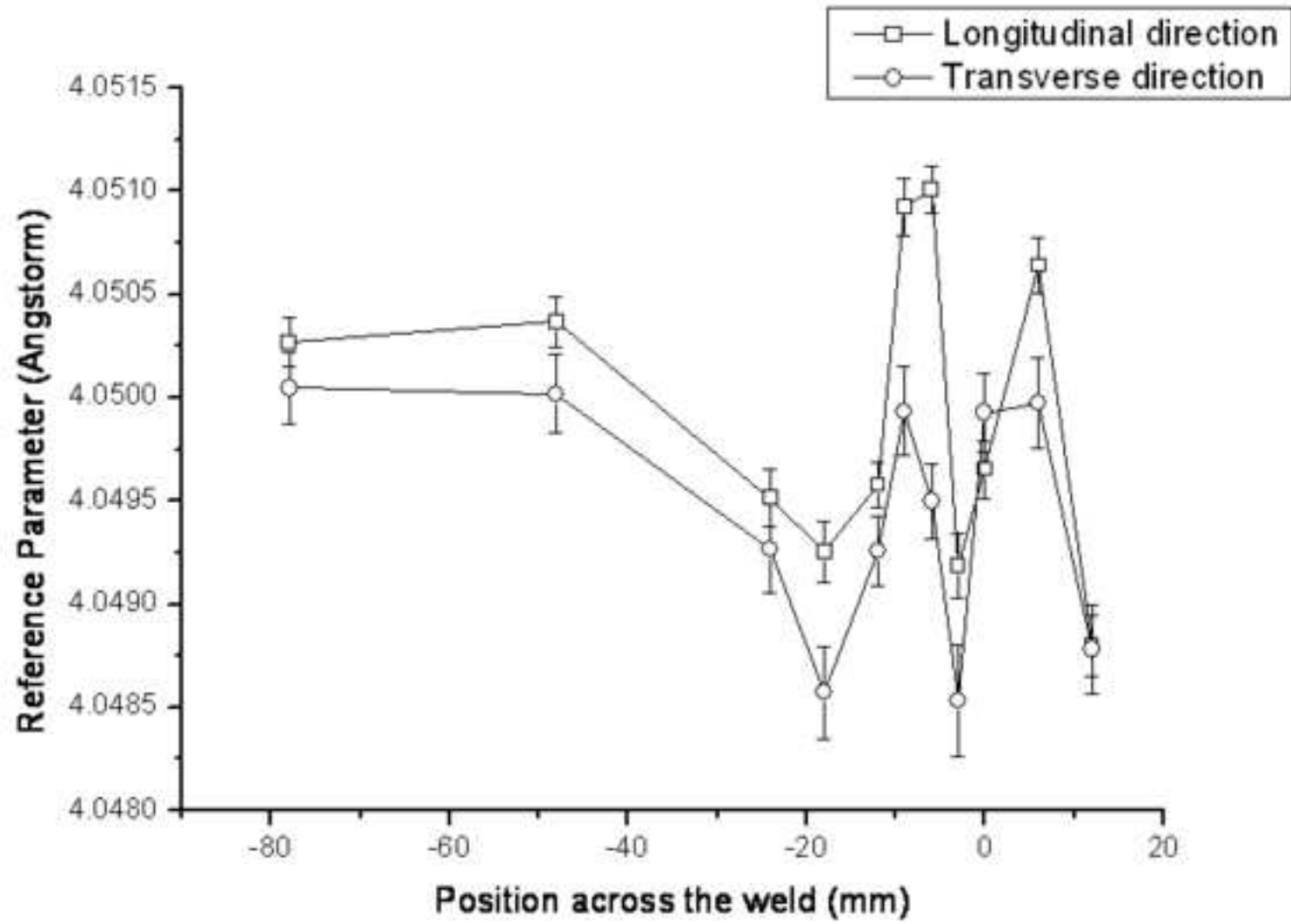


Figure 12

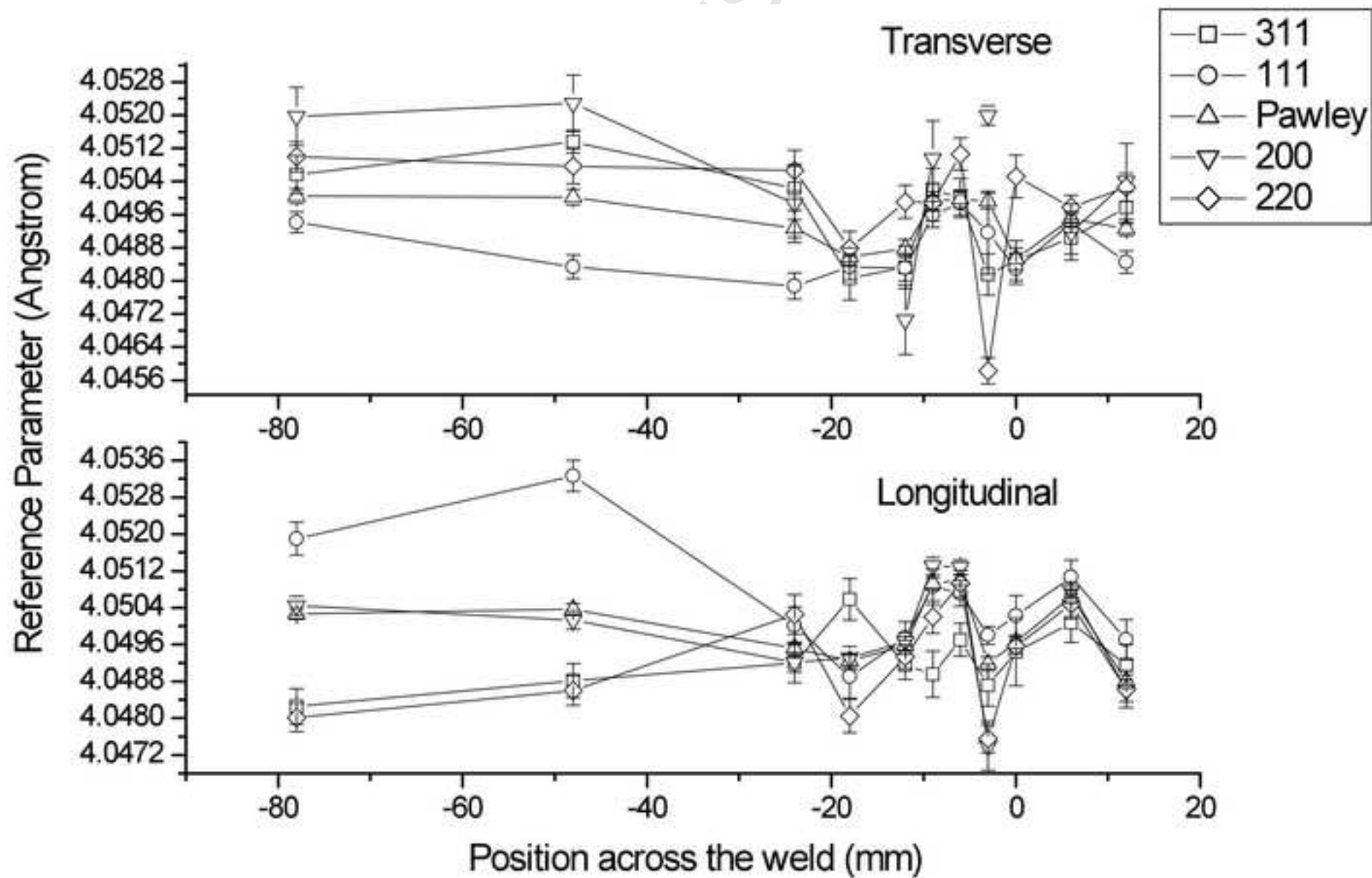


Figure 13

